इंटरनेट मानक

Disclosure to Promote the Right To Information

Whereas the Parliament of India has set out to provide a practical regime of right to information for citizens to secure access to information under the control of public authorities, in order to promote transparency and accountability in the working of every public authority, and whereas the attached publication of the Bureau of Indian Standards is of particular interest to the public, particularly disadvantaged communities and those engaged in the pursuit of education and knowledge, the attached public safety standard is made available to promote the timely dissemination of this information in an accurate manner to the public.

"जानने का अधिकार, जीने का अधिकार"
Mazdoor Kisan Shakti Sangathan
"The Right to Information, The Right to Live"

"पुराने को छोड नये के तरफ"
Jawaharlal Nehru
"Step Out From the Old to the New"

IS 1420 (1989): Light basic magnesium carbonate [CHD 1: Inorganic Chemicals]

"ज्ञान से एक नये भारत का निर्माण"
Satyanarayan Gangaram Pitroda
"Invent a New India Using Knowledge"

Act Number 22 of 2005
The Indian Parliament
RIGHT TO INFORMATION

Made Available By
Public.Resource.Org

"ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है"
Bhartṛhari—Nītiśatakam
"Knowledge is such a treasure which cannot be stolen"

BLANK PAGE

BRUTUM FULMEN
IGNORANTIA JURIS NON EXCUSAT
PROTECTED BY COPYRIGHT

IS 1420 : 1989
Reaffirmed 1999

Indian Standard

LIGHT BASIC MAGNESIUM CARBONATE — SPECIFICATION

(Second Revision)

भारतीय मानक

हल्के क्षारीय मैग्नेशियम कार्बोनेट — विशिष्टि

(दूसरा पुनरीक्षण)

First Reprint JANUARY 1999

UDC 661·846·622

© BIS 1990

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

March 1990

Price Group 6

Inorganic Chemicals (Miscellaneous) Sectional Committee, CDC 3

FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards on 31 July 1989, after the draft finalized by the Inorganic Chemicals (Miscellaneous) Sectional Committee had been approved by the Chemical Division Council.

This standard was first published in 1959 and revised in 1972. In this revision, requirements with respect to free moisture, loss on ignition, calcium oxide, iron, chloride, sulphate, heavy metals and arsenic contents have been amended. The requirement of matter insoluble in dilute hydrochloric acid has been deleted and a new requirement of carbon dioxide has been prescribed along with its method of test.

These changes have been brought out because the committee was convinced that the technology is now available in the country to prepare better quality of the product.

Light basic magnesium carbonate is one of the important raw materials for the rubber industry where it is used as a filler. To manufacture quality rubber products, the quality of light basic magnesium carbonate is of prime importance. The manufacture of very light grade material increases cost and also reduces manufacturing capacity. At present in rubber industry the product having a bulk density between 250 to 500 g/l is being used and hence two types are prescribed.

The largest portion of the production of light basic magnesium carbonate is used in the manufacture of heat insulating compositions. It is also used in preparing solid type rocket fuels and as an additive (1 to 2 percent) to table salt to make the salt free-flowing. It is also used in small quantities in tooth and face powders, polishing compounds and in pigments and paper.

Numerous references to the manufacturing of light basis magnesium carbonate of varying compositions can be found in the literature, but it has been shown that the conversion of the normal carbonate proceeds by way of the compounds $5\ MgO.4CO_2.6H_2O$ to the hydroxide, $Mg(OH)_2$ if boiling is continued long enough.

Light basic magnesium carbonate is prepared by direct carbonation of a slurry of magnesium hydroxide and by boiling the resulting $MgCO_3.3H_2O$. Sea water plants having relatively pure magnesium hydroxide slurries use this method. Flue gases of low carbon dioxide content can be used in the process because there is no separation step required and the hydroxide is converted directly into solid phase $MgCO_3.3H_2O$ or $5\ MgO.4CO_2.6H_2O$ depending on the temperature of carbonation.

In India small quantities of light basic magnesium carbonate are also manufactured by the reaction of sea water bitterns with soda ash.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

LIGHT BASIC MAGNESIUM CARBONATE — SPECIFICATION

(Second Revision)

1 SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for light basic magnesium carbonate.

2 REFERENCES

2.1 The Indian Standards listed below are necessary adjuncts to this standard:

IS No.	*Title*
IS 878 : 1975	Specification for graduated measuring cylinders (*first revision*)
IS 1070 : 1977	Specification for water for general laboratory use (*second revision*)
IS 2088 : 1983	Methods for determination of arsenic (*second revision*)
IS 4905 : 1968	Methods of random sampling

3 GRADES

3.1 There shall be three grades of the materials as follows:

a) *Grade* 1 — For use in the manufacture of 85 percent magnesia heat insulating compositions, in polishing compounds, pigments and paper.
b) *Grade* 2 — For use in the rubber industry.
c) *Grade* 3 — For use as an additive to table salt and in the manufacture of other fine chemicals.

4 TYPES

4.1 Each grade shall be of two types, namely, Type I and Type II depending upon its bulk density (*see* **5.2**).

5 REQUIREMENTS

5.1 Description

The material shall consist of light basic magnesium carbonate in the form of light, white, friable masses or powder, free from extraneous impurities and added substances.

5.2 Bulk Density

The bulk density of the material shall be as follows when determined by the method as prescribed in Annex A:

		Bulk Density g/l, *Max*
Grade 1	Type I	200
	Type II	300
Grade 2	Type I	200
	Type II	350
Grade 3	Type I	100
	Type II	140

5.3 Special Requirement for Rubber Industry

Grade 2 of the material shall pass such vulcanizing tests as agreed to between the purchaser and the supplier.

5.4 Fineness

Unless otherwise agreed to between the purchaser and the supplier the residue of the material shall be not more than 1·0 percent by mass, when tested according to the method prescribed in Annex B.

5.5 The material shall also comply with the requirements given in Table 1, when tested in accordance with the methods prescribed in Annex C. Reference to the relevant clauses of Annex C is given in col 6 of the table.

6 PACKING AND MARKING

6.1 Packing

The material shall be packed as agreed to between the purchaser and the supplier.

6.2 Marking

The packages shall be securely closed and legibly marked with the following information:

a) Name, grade and type of the material;
b) Mass of the material in the package;
c) Year of manufacture;
d) Manufacturer's name and/or his recognized trade-mark, if any; and
e) Batch number to enable the lot of manufacture to be traced back from records.

6.2.1 The packages may also be marked with the Standard Mark.

7 SAMPLING

7.1 The method of drawing representative samples of the material, number of tests to be performed and the criteria of conformity of the material to the requirements of this specification shall be as prescribed in Annex D.

Table 1 Requirement for Light Basic Magnesium Carbonate

(*Clause* 5.5)

Sl No.	Characteristic	Requirement			Method of Test Ref. to Cl No. in Annex C
		Grade 1	Grade 2	Grade 3	
(1)	(2)	(3)	(4)	(5)	(6)
i)	Free moisture, percent by mass, *Max*	1·10	1·10	1·10	C-2
ii)	Loss on ignition, percent by mass, *Max*	54·80	54·80	54·80	C-3
iii)	Calcium oxide (CaO), percent by mass, *Max*	0·2	0·2	0·2	C-4
iv)	Magnesium oxide (MgO), percent by mass	40 to 43.5	40 to 43·5	40 to 43·5	C-5
v)	Iron and aluminium (as oxides), percent by mass, *Max*	0·1	0·1	—	C-6
vi)	Iron (as Fe), percent by mass, *Max*	—	—	0·006	C-7
vii)	Chlorides (as Cl), percent by mass, *Max*	—	0·09	—	C-8
viii)	Sulphates (as SO_4), percent by mass, *Max*	—	0·02	—	C-9
ix)	Manganese (as Mn), percent by mass, *Max*	—	0·004	—	C-10
x)	Copper, percent by mass, *Max*	—	To pass the test	—	C-11
xi)	Heavy metals (as Pb), parts per million, *Max*	—	—	3	C-12
xii)	Arsenic (as As_2O_3), parts per million, *Max*	—	—	1	C-13
xiii)	Carbon dioxide, percent by mass, *Min*	37·27	36·27	36·27	C-14

ANNEX A

(*Clause* 5.2)

DETERMINATION OF BULK DENSITY

A-1 APPARATUS

A-1.1 Assemble the apparatus as shown in Fig. 1. The measuring cylinder *A* shall be of 250 ml capacity and shall conform to IS 878 : 1975.

The base of the measuring cylinder shall be ground flat. The distance between the flat-ground part of the base of the measuring cylinder *A* and the rubber base pad *B*, when the measuring cylinder *A* is raised to the full height, shall be 25 ± 2 mm.

A-1.2 The rubber base pad *B* shall have a Shore hardness of 42 to 50.

A-1.3 Pans of the balance shall be at least 10 cm in diameter and the balance shall have an accuracy of 0·1 g or better.

A-2 PROCEDURE

A-2.1 Dry-sieve about 20 g of the material through 250-micron IS sieve by lightly brushing a 25 mm wide and 12·5 mm thick good quality paint brush on to a tared glazed paper and weigh it accurately. Slip the powder gently and smoothly into the measuring cylinder, which should be held at 45° to the vertical, without knocking or squeezing. Assemble the apparatus as shown in Fig. 1. With the thumb and fingers of one hand, gently grasp the upper part of the cylinder, and within one second, lift it as far as 25 mm (*see* Fig. 1). Do not jerk the cylinder by knocking it against the upper stop. At the start of the next second, release the cylinder smoothly. Continue lifting and dropping at the rate of one drop per second until 50 complete drops have been given. Once every two seconds, a gently twist of about 10° should be given to the cylinder. As soon as 50 drops have been completed, raise the cylinder to eye level and read the volume of the sieved material.

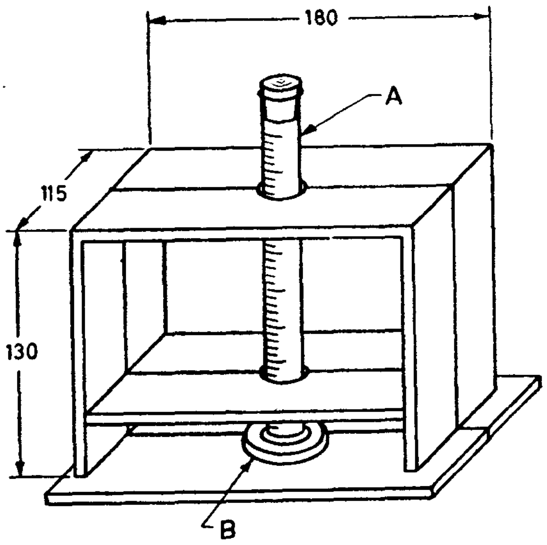
A-3 CALCULATION

$$\text{Bulk density, g/l} = \frac{M}{V}$$

where

M = mass in g of the sieved material taken for the test, and

V = final volume in litres of the sieved material.



All dimensions in millimetres.

FIG. 1 APPARATUS FOR THE DETERMINATION OF BULK DENSITY

ANNEX B

(*Clause* 5.4)

DETERMINATION OF FINENESS

B-1 APPARATUS

B-1.1 Gallie-Porritt Sieve

The apparatus (*see* Fig. 2) consists of a metal funnel terminating at the foot in a short cylindrical outlet, in which is inserted a shallow removable cup. To the bottom of the cup, a wire gauze equivalent to 75-micron IS Sieve of 25 mm diameter is soldered. Water under a pressure of 1·5 to 3·5 kgf/cm^2 is supplied by a tube fitted with a nozzle designed to discharge a spreading jet throughout the wire gauze, and the tube is so arranged that the distance of the orifice in relation to the wire gauze can be adjusted. The tube is provided with a filter to ensure removal of any solid particles from the water. A second tube which is also provided with a filter, is provided to supply a gentle stream of water for wetting the powder and for supplying more water to the funnel during the progress of the test.

B-1.1.1 The area of wire gauze shall be sufficiently large to prevent undue loss of pressure head in the case of first tube.

B-2 PROCEDURE

B-2.1 Weigh accurately about 50 g of the material. Half fill the funnel with water from the subsidiary supply, then bring the central jet (*see* Fig. 2) into operation. Transfer the weighed material to the metal funnel either dry or in the form of a slurry with a suitable wetting agent. Turn off the subsidiary supply and, if necessary, adjust the height of the central jet so that the level of water in the funnel commences to fall. When the level has fallen within 50 mm of the sieve, turn on subsidiary supply until the level is within 50 mm from the top of the funnel and maintain there until the liquid appears clear and then for a further period of one minute. Turn off the subsidiary supply and allow the level to fall again. When funnel is empty, turn off the main jet and allow to drain. Remove the cup and examine for any large soft agglomerates. If present, break up gently with the finger and replace the cup and wash with water from the subsidiary supply for 2 minutes. Transfer the grit on the sieve of watch-glass, dry at 100°C and weigh.

B-2.1.1 Powders which are very light are difficult to be wetted at a time in large quantity as 50 g. To facilitate wetting of these powders, either a small amount of wetting agent may be added or the material should be added in small quantities into the apparatus.

B-3 CALCULATION

Express the mass of residue as percentage of mass of the material taken for the test.

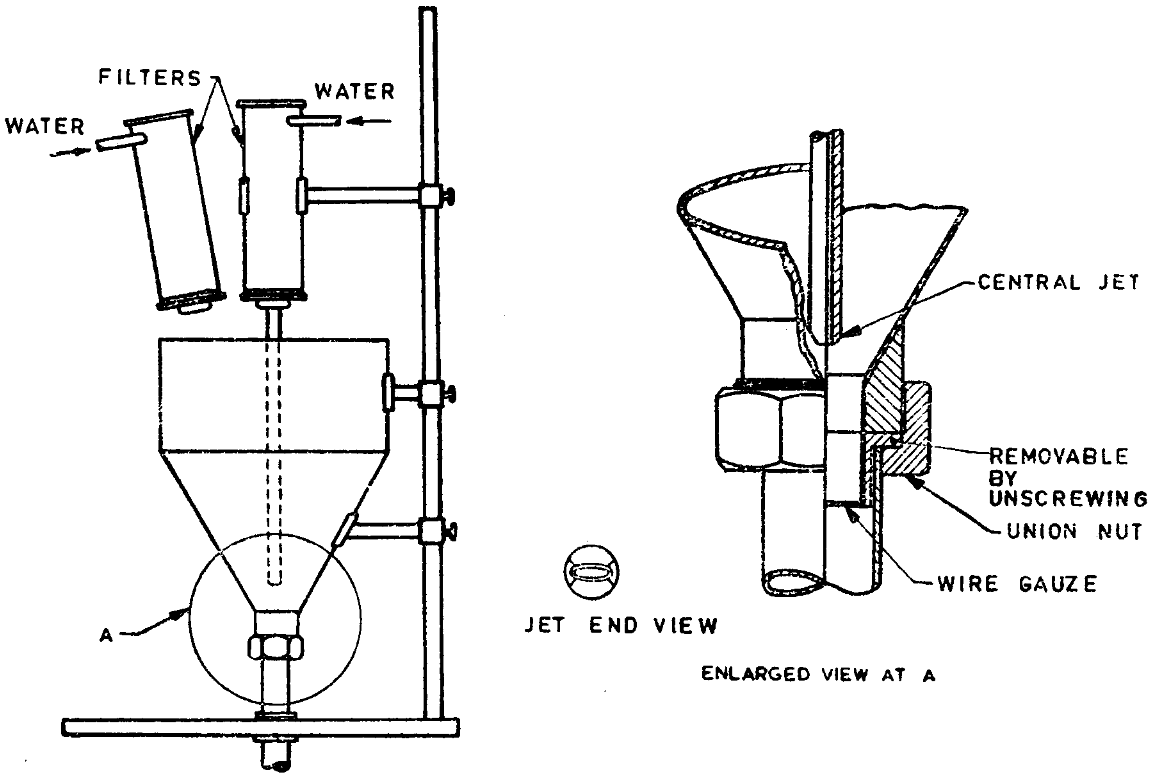


FIG. 2 GALLIE-PORRITT SIEVE FOR DETERMINATION OF FINENESS

ANNEX C

(*Clause* 5.5)

METHODS OF TEST FOR LIGHT BASIC MAGNESIUM CARBONATE

C-1 QUALITY OF REAGENTS

C-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 : 1977) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

C-2 DETERMINATION OF FREE MOISTURE

C-2.1 Procedure

Weigh accurately about 5 g of the material in a weighed, clean and dry petri dish. Keep in a vacuum desiccator over concentrated sulphuric acid for 24 hours and weigh the dish again.

C-2.2 Calculation

$$\text{Moisture, percent by mass} = 100 \times \frac{M_1}{M}$$

where

M_1 = loss in mass in g, and

M = mass in g of the material taken for the test.

C-3 DETERMINATION OF LOSS ON IGNITION

C-3.1 Procedure

Weigh accurately about 1 g of the material in a tared crucible, previously heated to constant mass in a muffle furnace. Place the crucible along with its contents in the furnace maintained at a temperature of not less than 900°C and ignite. Cool in a desiccator and weigh. Repeat the operation till constant mass is obtained.

C-3.2 Calculation

$$\text{Loss on ignition, percent by mass} = 100 \times \frac{M_1}{M}$$

where

M_1 = loss in mass in g, and

M = mass in g of the material taken for test.

C-4 DETERMINATION OF CALCIUM OXIDE

Calcium oxide may be determined either by the permanganate method or EDTA method. In case of dispute, the permanganate method shall be considered as the referee method.

C-4.1 Permanganate Method

C-4.1.1 *Reagents*

C-4.1.1.1 *Dilute hydrochloric acid*, approximately 5 N.

C-4.1.1.2 *Ammonium oxalate solution*, approximately 5 percent.

C-4.1.1.3 *Dilute ammonium hydroxide*, approximately 5 N and 1 N.

C-4.1.1.4 *Methyl red indicator*, 0·1 g in 100 ml of rectified spirit.

C-4.1.1.5 *Oxalic acid solution*, saturated in water.

C-4.1.1.6 *Dilute potassium permanganate solution*, approximately 0·1 percent (*m*/*v*).

C-4.1.1.7 *Dilute sulphuric acid*, approximately 5 N.

C-4.1.1.8 *Standard potassium permangante solution*, approximately 0·05 N.

C-4.1.2 *Procedure*

Dissolve about 5 g of the material, accurately, weighed, in about 60 ml of dilute hydrochloric acid. Heat to near boiling and add ammonium hydroxide (5 N) dropwise to make the solution alkaline. If necessary, add a few more drops of methyl red indicator. Add oxalic acid solution drop by drop until the indicator becomes just red. While keeping the solution near boiling, add about 20 ml of ammonium oxalate solution slowly with stirring. Allow the precipitate to stand on a boiling water-bath for 30 minutes and then decant the supernatant solution through a filter paper (Whatman No. 42 or equivalent). There is no need to transfer the precipitate to the filter paper. Dissolve the precipitate remaining in the beaker in minimum quantity of dilute hydrochloric acid by heating to boiling and add dilute ammonium hydroxide (5 N) dropwise until a precipitate begins to form. Dilute to about 100 ml, heat to near boiling and add about 10 ml of ammonium oxalate solution slowly with stirring. Allow to stand on a boiling water-bath for 2 hours.

C-4.1.2.1 Transfer the precipitate to the filter paper. Wash the precipitate first with dilute ammonium hydroxide solution (1 N) and then with cold water until the filtrate does not decolourise a hot dilute potassium permanganate solution (0·1 percent) in the presence of dilute sulphuric acid. Puncture the filter paper and transfer the precipitate to a beaker with a fine jet of hot water. Wash the filter paper five times with hot dilute sulphuric acid using 10 ml of the dilute acid each time, collecting the washings into the beaker. Heat the contents of the beaker to 60°C. Titrate the solution with standard potasium permanganate solution.

C-4.1.3 *Calculation*

$$\text{Calcium oxide (CaO), percent by mass} = \frac{2{\cdot}804\, V N}{M}$$

where

V = volume in ml of standard potassium permanganate solution used in titration,

N = normality of standard potassium permanganate solution used, and

M = mass in g of the material taken for the test.

C-4.2 EDTA Method

C-4.2.1 *Reagents*

C-4.2.1.1 *Dilute hydrochloric acid*, approximately 5 N.

C-4.2.1.2 *Potassium hydroxide solution*

Approximately 10 N. Before use, shake it to break any film of carbonate which may form on its surface. Allow it to settle and then take out the solution by decantation or, if suspended carbonate particles are still seen, by filtration through glass wool.

C-4.2.1.3 *Standard calcium solution*

0·01 M. Weigh 1·001 g of calcium carbonate and dissolve in minimum quantity of dilute hydrochloric acid. Make up the volume with water to 1 litre.

C-4.2.1.4 *Patton and Readers' indicator*

Mix 0·1 g of 2 hydroxy 1-(2-hydroxy-4-sulpho-1-napthylazo)-3-naphthoic acid with 10 g of potassium chloride and grind into a fine mixture.

C-4.2.1.5 *Standard EDTA solution*

Weigh 3·72 g of disodium ethylene diamine tetraacetate dihydrate (EDTA) and dissolve in water. Make up the volume to one litre, and standardize it as follows:

> Take 10 ml of standard calcium solution in a conical flask. Add 20 to 30 ml of water and boil to expel any carbon dioxide. Add 1 to 2 ml of potassium hydroxide solution and cool slightly. Add about 0·2 g of the indicator and titrate with EDTA solution until the colour changes from wine red to pure blue without any tinge of violet.

$$\text{Molarity of EDTA solution} = \frac{10\, M_1}{V_2}$$

where

M_1 = molarity of standard calcium solution, and

V_2 = volume of EDTA solution required for titration.

C-4.2.1.6 *Hydroxylamine hydrochloride*, solid.

C-4.2.2 *Procedure*

Dissolve 5 g of the material, accurately weighed, in about 50 to 60 ml of dilute hydrochloric acid and boil to expel all carbon dioxide. Cool and make up to 250 ml in a volumetric flask. Mix well and pipette out two portions of 50 ml each, in two conical flasks. Reserve the balance of solution for determination of magnesium oxide.

C-4.2.2.1 To one flask add 4 to 5 ml of potassium hydroxide solution and then about 50 ml of water. Add about 0·2 g of indicator and quickly titrate roughly against standard EDTA solution until the colour changes to violet blue. Note the volume of titrant consumed and add this volume directly to the second flask. Heat this to boiling, add 4 to 5 ml of potassium hydroxide solution and about 0·2 g of hydroxylamine hydrochloride and boil for a few seconds. Cool by adding about 50 ml of previously boiled and cooled water. Add about 0·2 g of hydroxylamine indicator and titrate carefully, shaking after each addition of titrant, until no further change of colour occurs and the test solution is clear blue without any tinge of violet. The titre is the total volume of EDTA solution added to the second flask.

C-4.2.3 *Calculation*

$$\text{Calcium oxide (CaO), percent by mass} = \frac{14{\cdot}02\, V M_2}{M}$$

where

V = volume in ml of standard EDTA solution used for the titration,

M_2 = molarity of the EDTA solution used, and

M = mass in g of the material taken for the test.

C-5 DETERMINATION OF MAGNESIUM OXIDE (MgO)

Magnesium oxide is determined either gravimetrically by pyrophosphate method or by EDTA method. In case of dispute, the gravimetric method shall be considered as the referee method.

C-5.1 Gravimetric Method

C-5.1.1 *Reagents*

C-5.1.1.1 *Ammonium oxalate solution,* approximately 5 percent (*m*/*v*).

C-5.1.1.2 *Dilute ammonium hydroxide,* approximately 5 N and 1 N.

C-5.1.1.3 *Concentrated nitric acid*

C-5.1.1.4 *Concentrated hydrochloric acid*

C-5.1.1.5 *Diammonium hydrogen phosphate solution* approximately 25 percent.

C-5.1.1.6 *Phenolphthalein indicator,* approximately 25 percent.

C-5.1.2 *Procedure*

Pipette out 10 ml of the solution reserved under C-4.2.2. Add water to make the volume to about 100 ml. Heat the solution and boil for about 10 minutes. Add 10 ml of ammonium oxalate solution. To the hot solution, constantly stirred, add dilute ammonium hydroxide solution (5 N) drop by drop until the mixture is strongly alkaline. Let it stand for 2 hours. Decant the clear supernatant liquid through a filter paper. Transfer the precipitate to the filter paper and wash it with dilute ammonium hydroxide solution (1 N) and then with cold water. Take the filtrate and the washings in an evaporating dish and evaporate to dryness. To the residue, add 15 ml of concentrated nitric acid and evaporate again to dryness. To the residue, again add 2 to 3 ml of concentrated nitric acid and again evaporate to dryness. Cool and dissolve the residue in concentrated hydrochloric acid. Dilute with water to 100 ml, add 10 to 15 ml of diammonium hydrogen phosphate solution and 2 to 3 drops of phenolphthalein indicator. Heat to boiling and add dilute ammonium hydroxide (5 N) until a light pink colour appears and slight precipitate forms. Cool and stir and when the precipitate becomes distinctly crystalline, add more dilute ammonium hydroxide (5 N) until a deep pink colour is obtained. Set aside and allow the precipitate to stand overnight. Filter, and then wash the precipitate with dilute ammonium hydroxide (1 N) until the washings are free from chloride. Place the filter paper with its contents in a tared platinum crucible. Dry in an air oven, char the filter paper by gently heating over a low flame and then slowly increase the flame and finish by heating at 900 to 950°C till the precipitate is almost white. Cool the crucible in a desiccator and weigh. Repeat the process till constant mass is obtained.

C-5.1.3 *Calculation*

$$\text{Magnesium oxide (MgO), percent by mass} = \frac{905{\cdot}2\, M_1}{M}$$

where

M_1 = mass in g of the precipitate obtained, and

M = mass in g of the material taken for the test under C-4.2.2.

C-5.2 EDTA Method

C-5.2.1 *Reagents*

C-5.2.1.1 *Eriochrome black T indicator*

Dissolve 0·1 g of eriochrome black T indicator in 25 ml of methyl alcohol.

C-5.2.1.2 *Ammonium hydroxide-ammonium chloride buffer solution*

Mix 350 ml of ammonium hydroxide solution (r.d. 0·90) and 54 g of ammonium chloride. Dilute with water and make up the volume to 1 000 ml. The *p*H of the solution will be more than 10.

C-5.2.1.3 *Standard EDTA solution,* same as under **C-4.2.1.5.**

C-5.2.2 *Procedvre*

Pipette out 20 ml of the solution prepared under **C-4.2.2** into a 1 litre graduated flask and dilute to the mark. Pipette out 20 ml of this diluted solution into a 250 ml conical flask, add 20 ml of water, 1 ml of eriochrome black T indicator and 25 ml of ammonium hydroxide-ammonium chloride buffer solution. Heat to 40 to 50°C and then titrate against standard EDTA solution until the colour changes from wine red to distinct blue. The temperature of the solution should not be allowed to fall below 40°C during titration.

C-5.2.3 *Calculation*

$$\text{Magnesium oxide (MgO), percent by mass} = \frac{1\,260\,(V_1 - 0{\cdot}006\,V)\,M_2}{M}$$

where

V_1 = volume in ml of EDTA solution used in the titration in **C-5.2.2,**

V = volume in ml of EDTA solution used in the titration in **C-5.2.2,**

M_2 = molarity of the EDTA solution used, and

M = mass in g of the material taken for the test in **C-4.2.2.**

C-6 DETERMINATION OF IRON AND ALUMINIUM

C-6.1 Reagents

C-6.1.1 *Concentrated Hydrochloric Acid*

C-6.1.2 *Dilute Ammonium Hydroxide,* approximately 10 percent (*m*/*v*).

C-6.1.3 *Methyl Red Indicator Solution,* dissolve 0·1 g of methyl red in 100 ml of rectified spirit.

C-6.1.4 *Ammonium Chloride Solution,* pproximately 2 percent (*m*/*v*).

C-6.1.5 *Dilute Hydrochloric Acid,* approximately 10 percent (*v*/*v*).

C-6.1.6 *Ammonium Nitrate Solution,* approximately 2 percent (*m*/*v*).

C-6.2 Procedure

Weigh accurately about 2 g of the material and dissolve in a mixture of 5 ml of concentrated hydrochloric acid and 75 ml of water, boil and make alkaline with dilute ammonium hydroxide solution using methyl red as indicator. Boil for 1 minute, filter, wash and then precipitate with hot ammonium chloride. Dissolve the precipitate as completely as possible by passing 20 ml of hot dilute hydrochloric acid through the filter paper and wash the filter paper with sufficient hot water to adjust the volume of the solution to 50 ml. Boil the solution and make alkaline with dilute ammonium hydroxide solution using methyl red as indicator. Boil for one minute, filter through the same filter paper and wash the precipitate with hot ammonium nitrate solution. Dry and ignite to constant mass at a temperature between 800 and 1 000°C.

C-6.3 Calculation

$$\text{Iron and aluminium (as oxides), percent by mass} = \frac{100\,A}{M}$$

where

A = mass in g of the ignited residue, and

M = mass in g of the material taken for the test.

C-7 DETERMINATION OF IRON

C-7.1 Apparatus

C-7.1.1 *Nessler Cylinder,* 50 ml capacity.

C-7.2 Reagents

C-7.2.1 *Dilute Nitric Acid,* 1 : 9 (*v*/*v*).

C-7.2.2 *Ammonium Persulphate,* Solid.

C-7.2.3 *Butanolic Potassium Thiocyanate Solution*

Dissolve 10 g of potassium thiocyanate in 10 ml of water. Add sufficient *n*-butanol, make up to 100 ml and shake vigorously until the solution is clear.

C-7.2.4 *Standard Iron Solution*

Dissolve 0·702 0 g of ferrous ammonium sulphate [$FeSO_4(NH_4)_2SO_4{\cdot}6H_2O$] in 10 ml of dilute sulphuric acid (10 percent *v*/*v*) and dilute with water to 1 000 ml. Pipette out 10 ml of this solution and dilute with water to make up the volume to 100 ml. One millilitre of this solution contains 0·01 mg of iron (as Fe).

C-7.3 Procedure

Weigh exactly 100 mg of the material and boil with 5 ml of dilute nitric acid for 1 minute. Cool, dilute to 50 ml with water, add about 50 mg of ammonium persulphate and 5 ml of butanolic ammonium thiocyanate solution, mix and transfer to a Nessler cylinder. Treat in the same manner exactly 2 ml of standard iron solution with the same quantities of the reagents. Dilute both the solutions to the mark and mix.

C-7.3.1 The limit prescribed in Table 1 shall be considered to have not exceeded if the intensity of the red colour produced in the butanolic layer in the test with the material is not greater than that produced in the control test.

C-8 DETERMINATION OF CHLORIDES

C-8.1 Apparatus

C-8.1.1 *Nessler Cylinder*, 50 ml capacity.

C-8.2 Reagents

C-8.2.1 *Concentrated Nitric Acid*

C-8.2.2 *Standard Sodium Chloride Solution*

Dissolve 1·649 g of sodium chloride in water and make up the volume to 1 000 ml. Pipette out 10 ml of the solution, dilute with water and make up the solution to 100 ml. One millilitre of this solution contains 0·1 mg of chloride (as Cl).

C-8.2.3 *Silver Nitrate Solution*, approximately 2 percent (*m*/*v*).

C-8.3 Procedure

Dissolve 1·00 g of the material in water by the addition of 5 ml of concentrated nitric acid and boil cautiously to remove carbon dioxide. Cool and make up the volume to 100 ml. Pipette out 10 ml of this solution into a Nessler cylinder. Add 1 ml of silver nitrate dilute to 50 ml mark. Carry out a control in the other Nessler cylinder using 2 ml of standard sodium chloride solution and the same quantities of other reagents and finally diluting to 50-ml mark. Stir both the solutions with glass rods and compare the turbidity-produced in the two cylinders after 5 minutes.

C-8.3.1 The limit prescribed shall be taken as not having been exceeded if the turbidity produced in the test with the material is not greater than that produced in the control test.

C-9 DETERMINATION OF SULPHATES

C-9.1 Apparatus

C-9.1.1 *Nessler Cylinder*, 50 ml capacity.

C-9.2 Reagents

C-9.2.1 *Dilute Hydrochloric Acid*, approximately 5 N.

C-9.2.2 *Standard Sulphate Solution*

Dissolve 0·131 4 g of potassium sulphate in water and make up to the volume to 1 000 ml. Take 10 ml of this solution and dilute it further to 100 ml. One millilitre of the final solution is equivalent to 0·01 mg of sulphate (as SO_4).

C-9.2.3 *Barium Chloride Solution*, approximately 10 percent (*m*/*v*).

C-9.3 Procedure

Dissolve 0·50 g of the material in water by the addition of 3 ml of dilute hydrochloric acid and boil continuously to remove carbon dioxide. Cool and make up the volume to 100 ml. Transfer by means of a pipette 10 ml of this solution to a Nessler cylinder. Add 1 ml of barium chloride solution. Stir the mixture and add water to make up the volume to 50 ml. Carry out a control in the other Nessler cylinder using 10 ml of standard sulphate solution, and the same quantities of other reagents and finally diluting to 50 ml. Compare the turbidity produced in the two cylinders after 5 minutes.

C-9.3.1 The limit prescribed shall be considered as not having been exceeded if the turbidity produced in the test with the material is not greater than that produced in the control test.

C-10 DETERMINATION OF MANGANESE

C-10.1 Apparatus

C-10.1.1 *Nessler Cylinder*, 50 ml capacity.

C-10.2 Reagents

C-10.2.1 *Dilute Sulphuric Acid*, approximately 5 N.

C-10.2.2 *Concentrated Nitric Acid*

C-10.2.3 *Concentrated Sulphuric Acid*

C-10.2.4 *Phosphoric Acid*, 85 percent by mass.

C-10.2.5 *Standard Potassium Permanganate Solution*

Prepare dilute solution of potassium permanganate by diluting to 500 ml in a volumetric flask, a quantity of a freshly standardized solution in accordance with the following formula:

$$A = \frac{0{\cdot}455\,1}{N}$$

where

A = volume in ml of the standard potassium permanganate solution to be made up to a volume of 500 ml, and

N = normality of the standard potassium permanganate solution.

One millilitre of this solution is equivalent to 0·01 mg of manganese (Mn).

NOTE — Do not keep the solution for more than one week.

C-10.2.6 *Potassium Periodate*

C-10.3 Procedure

Weigh accurately 1·00 g of the material. Moisten with a few millilitres of water and add about 30 ml of dilute sulphuric acid. Evaporate to dryness. Add a mixture of 1 ml of concentrated nitric acid and 6 ml of concentrated sulphuric acid. Heat on a sand bath till fumes of sulphur trioxide start evolving. Dilute with water and filter the solution into a beaker. Add 10 ml of phosphoric acid and about 0·5 g of potassium periodate. Boil for 5 minutes, cool slightly and add again about 0·5 g of potassium periodate and boil. When the colour appears to have developed to the maximum, place the beaker on a steam-bath for about 15 minutes. Cool the solution and transfer it to a Nessler cylinder, and dilute with water to 50 ml. Carry out a control test using 4 ml of standard potassium permanganate solution in the other Nessler cylinder.

C-10.3.1 The limit prescribed in Table 1 shall be considered as not having been exceeded if the colour produced with the material is not greater than that produced in the control test.

C-11 TEST FOR COPPER

C-11.1 Reagents

C-11.1.1 *Dilute Hydrochloric Acid,* approximately 5 N.

C-11.1.2 *Dilute Ammonium Hydroxide,* approximately 10 percent (*m/v*).

C-11.2 Procedure

Dissolve about 10 g of the material in about 30 ml of dilute hydrochloric acid and boil to remove carbon dioxide. Add dilute ammonium hydroxide to make the solution distinctly alkaline.

C-11.2.1 The material shall be considered to have satisfied the requirements of the test if no blue colour develops.

C-12 DETERMINATION OF HEAVY METALS

C-12.1 Apparatus

C-12.1.1 *Nessler Cylinder.* 50 ml capacity.

C-12.2 Reagents

C-12.2.1 *Dilute Hydrochloric Acid,* approximately 5 N.

C-12.2.2 *Dilute Acetic Acid*

1 N approximately, prepared by diluting 60 ml of glacial acetic acid with sufficient water to make 1 000 ml.

C-12.2.3 *Sodium Sulphide Solution,* 10 percent (*m/v*).

C-12.2.4 *Standard Lead Solution*

Dissolve 159·8 mg of lead nitrate in 100 ml of water to which has been added 1 ml of concentrated nitric acid, and then dilute with water to exactly 1 000 ml. Take 10 ml of this solution in a 100 ml volumetric flask and dilute to the mark with water. One millilitre of this solution is equivalent to 0·01 mg of lead (Pb). The diluted solution shall be freshly prepared.

C-12.3 Procedure

Dissolve 1·00 g of the material in 10 ml of dilute hydrochloric acid, and evaporate the solution on a steam-bath to dryness. Towards the end of the evaporation, stir frequently to disintegrate the residue so that finally a dry powder is obtained. Dissolve the residue in 20 ml of water and evaporate to dryness in the same manner as before. Redissolve the residue in 20 ml of water, filter, if necessary, and add to the filtrate 2 ml of dilute acetic acid and water to make 25 ml.

Transfer the solution to a Nessler cylinder and add 10 ml of sodium sulphide solution. Take in another Nessler cylinder 2 ml of dilute acetic acid and 3 ml of standard lead solution. Dilute both the solutions with water to the mark, mix well and allow to stand for 10 minutes. Compare the colour produced in the two cylinders.

C-12.3.1 The limit prescribed in Table 1 shall be considered to have not exceeded if the colour produced by the material is not deeper than that produced in the control test.

C-13 DETERMINATION OF ARSENIC

C-13.1 Procedure

Dissolve 1·00 g of the material in 5 ml of dilute hydrochloric acid (5 N), and carry out the test for arsenic as prescribed in IS 2088 : 1983 using for comparison a stain obtained with 0·005 mg of arsenic trioxide (as As_2O_3).

C-14 DETERMINATION OF CARBON DIOXIDE

C-14.1 Principle

The action of phosphoric acid on light basic magnesium carbonate results in the evolution of carbon dioxide which is absorbed over ascarite (sodium hydroxide-asbestos). The increase in the weight of ascarite determines the amount of carbon dioxide evolved from a known mass of the sample.

C-14.2 Apparatus

The apparatus used for the gravimetric determination of carbon dioxide is shown in Fig. 3. *C* is a wide-mouthed round-bottomed flask of 100 ml capacity. *B* is a dropping funnel containing syrupy phosphoric acid to which is attached a U-tube. A containing ascarite to absorb atmospheric carbon dioxide. *D* is a bubbler containing syrupy phosphoric acid. This is attached to a U-tube *E* containing magnesium perchlorate. The U-tubes *F* and *G* contain ascarite. One third of these tubes are filled with the desiccant magnesium perchlorate (anhydrous) to absorb moisture. Absorption of carbon dioxide on ascarite results in release of water due to the reaction of carbon dioxide with sodium hydroxide. The moisture is allowed to U-tubes *F* and *G* will affect the result of carbon dioxide value. The U-tube *H* also contains magnesium perchlorate. *J* is another bubbler containing concentrated sulphuric acid to absorb moisture.

C-14.3 Reagents

C-14.3.1 *Syrupy Phosphoric Acid*

C-14.3.2 *Magnesium Perchlorate, Anhydrous,* 1·1 to 7·10-micron IS Sieve.

C-14.4 Procedure

Assemble the apparatus as shown in Fig. 3. Suspend the U-tubes by metallic wire on a metallic rod. Make all the joints with short lengths of stout walled rubber tubing, making sure that the two ends of glass tubes are in contact with each other. Ensure that the apparatus is gas-tight.

C-14.4.1 Weigh accurately 0·5 g of the sample and place in the clean and dry round bottomed flask *C*. Remove the two ascarite containing U-tubes, *F* and *G*, wipe them with clean linen and then weigh.

C-14.4.2 Replace the flask and U-tubes to their original position and add 25 ml of syrupy phosphoric acid into the flask *C* through the dropping funnel *B*. Close the tap of the funnel. Heat the flask gently till the decomposition of the sample. Regulate the temperature so that no more than 2 bubbles of the gas pass through the bubbler. The aspirator tap should also be accordingly adjusted to maintain the gas flow rate at 2-3 bubbles per second.

C-14.4.3 Open the tap of the funnel and draw air through aspirator tap at the rate of about 2 bubbles per second for 5-10 minutes. Remove the U-tubes containing ascarite and weigh them.

C-14.4.4 Run reagent blank through all the steps mentioned above without taking the sample and weigh the ascarite containing U-tubes. This should be deducted from the mass of the U-tubes obtained with the sample.

C-14.4.5 *Calculation*

$$\text{Carbon dioxide, percent by mass} = \frac{(M_1 - M_2 - M_3)}{M} \times 100$$

where

M_1 = mass in g of U-tubes after the test,

M_2 = mass in g of U-tube before test,

M_3 = mass in g of U-tube in blank tests, and

M = mass in g of the sample taken for the test.

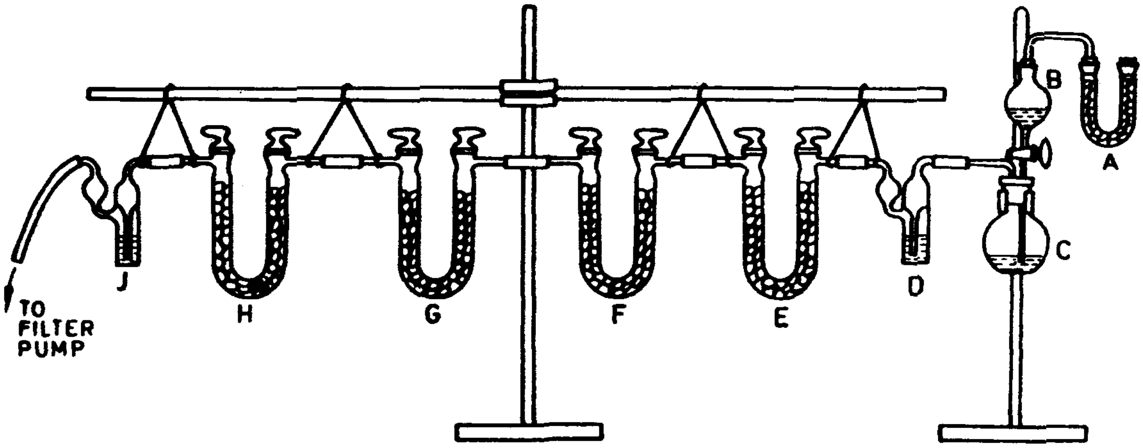


FIG. 3 APPARATUS FOR THE DETERMINATION OF CARBON DIOXIDE

ANNEX D

(Clause 7.1)

SAMPLING OF LIGHT BASIC MAGNESIUM CARBONATE

D-1 GENERAL REQUIREMENTS OF SAMPLING

D-1.0 In drawing and storing samples, the following precautions and directions shall be observed.

D-1.1 Samples shall not be taken in an exposed place.

D-1.2 The sampling implement and sample containers shall be clean and dry.

D-1.3 After filling, sample containers shall be closed airtight and marked with all details of sampling.

D-2 SCALE OF SAMPLING

D-2.1 Lot

In any consignment, material of the same grade, type and produced in one batch of manufacture shall constitute a lot.

D-2.2 Selection of Sample

For checking the conformity of the lot to the requirements of this specification tests shall be carried out for each lot separately. The number of containers to be selected for this purpose shall be as given in Table 2.

The containers to be included in the sample shall be selected at random from the lot with the help of random number table. For random selection procedures guidance may be had from IS 4905 : 1968.

D-3 NUMBER OF TESTS AND CRITERIA FOR CONFORMITY

D-3.1 For each of the selected containers draw with appropriate sampling instrument about 300 g of light basic magnesium carbonate. The portions from each container shall be transferred to separate sample bottles. These are known as individual samples.

D-3.2 From each of the individual samples equal amount of material shall be collected and mixed well to form a composite sample weighing 300 g.

D-3.3 Tests for magnesium oxide content shall be conducted on individual samples. Tests for all the other characteristics shall be made on the composite sample.

D-3.4 For declaring the conformity of the lot to the requirements of this specification, test results of individual samples as well as composite sample shall satisfy the requirements given in Table 1.

Table 2 Number of Containers to be Selected in the Sample

(*Clause* D-2.2)

Lot Size (Number of Containers in the Lot) (1)	Sampling Size (Number of Containers to be Selected) (2)
Up to 10	3
11 to 25	4
26 to 50	5
51 to 100	6
101 and above	7

Bureau of Indian Standards

BIS is a statutory institution established under the *Bureau of Indian Standards Act*, 1986 to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

Copyright

BIS has the copyright of all its publications. No part of these publications may be reproduced in any form without the prior permission in writing of BIS. This does not preclude the free use, in the course of implementing the standard, of necessary details, such as symbols and sizes, type or grade designations. Enquiries relating to copyright be addressed to the Director (Publication), BIS.

Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Handbook' and 'Standards Monthly Additions'.

This Indian Standard has been developed from Doc: No. **CDC 3 (9417)**

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones: 323 01 31, 323 33 75, 323 94 02

Telegrams: Manaksanstha
(Common to all offices)

Regional Offices:

		Telephone
Central	: Manak Bhavan, 9 Bahadur Shah Zafar Marg NEW DELHI 110002	323 76 17, 323 38 41
Eastern	: 1/14 C.I.T. Scheme VII M, V.I.P. Road, Maniktola CALCUTTA 700054	337 84 99, 337 85 61 337 86 26, 337 91 20
Northern	: SCO 335-336, Sector 34-A, CHANDIGARH 160022	60 38 43 60 20 25
Southern	: C.I.T. Campus, IV Cross Road, CHENNAI 600113	235 02 16, 235 04 42 235 15 19, 235 23 15
Western	: Manakalaya, E9 MIDC, Marol, Andheri (East) MUMBAI 400093	832 92 95, 832 78 58 832 78 91, 832 78 92

Branches : AHMADABAD. BANGALORE. BHOPAL. BHUBANESHWAR. COIMBATORE. FARIDABAD. GHAZIABAD. GUWAHATI. HYDERABAD. JAIPUR. KANPUR. LUCKNOW. NAGPUR. PATNA. PUNE. THIRUVANANTHAPURAM.

Printed by Reprography Unit, BIS, New Delh